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Founded September, 1928

Comprising
"The Paint and Varnish Record"
Founded in 1911

Entered as Second-class matter, May, 1935, at the Post Office at Washington, D. C. Under the Act of March 3, 1879.

Published the First of Every Month by Paint and Varnish PRODUCTION MANAGER

Member, Oil and Colour Chemists' Association, London, England.

M. R. DRAKE, *Publisher*
EDWARD R. DRAKE, *Editor*
Publication Office
Mills Building, Washington 6, D. C.

DUNCAN P. MACPHERSON
Advertising Manager
700 So. Washington Square
Philadelphia, Pa.

See Standard Rate and Data Service for rates. Advertising copy expected the 10th of each month.

Subscription price per year in the United States, $3.00; Canada and other Foreign Countries, $3.60; payable in advance.

INFLUENCE OF THE PHYSICAL-CHEMICAL PROPERTIES OF PIGMENTS ON ANTI-CORROSION PAINT FILMS.

By DR. HANS WAGNER *†

This article by a well known chemist is submitted without any responsibility on the part of this publication as to actual facts. Editor.

In a previous paper of the author on the MECHANISM OF PIGMENTATION (published in the PVPM) the present day knowledge on the properties required for anti-corrosion pigments was concentrated into eight guiding principles which are to serve as fundamental motives for the various points of importance treated in this present paper.

We are particularly interested in the following problems:

Preference must be given the lead pigments reacting with fatty oils. The chances to get away from the lead pigments entirely and to find high-grade specific anti-rusting pigments of entirely different compositions are less promising than the chances of obtaining satisfactory pigment mixtures consisting of relatively small amounts of active lead pigments and larger percentages of suitable auxiliary pigments.

It is also possible that satisfactory results can be obtained with plated pigments affording a corresponding saving of corrosion-active substances but it is still a matter of conjecture whether faultness plating of this type can be produced at all. It is to be assumed that the structure of the films with regard to the pigment arrangement and the formation of strengthening and stress-compensating products of reaction is more important than the electrochemical effects of the pigments. Special attention must be paid to the water contents of pigments and vehicles as well as to the importance of packing and shapes of pigments and of the influence of faulty spots within the paint films.

II. Basic Experiments

(a) Pigments arrangement within the films.

Several different anti-rusting or auxiliary pigments were ground in by three different methods: (1) The pigment powders were thrown into the oil and stirred slightly, (2) the oil was poured into the pigment and stirred slightly, (3) Pigments and oil were stirred together intensively. The pigment concentrations in these mixtures were adapted partly to those common with the usual brushable paints, while other mixtures contained only sufficient pigments to render microscopic examination of the films (applied to glass plates) in transmitted light just possible. The pigments employed were: red lead red oxide of iron, zinc chormate, aluminum powder, iron mica

*† Professor Dr. Hans Wagner, Dean of the Institute for Paint Technology at the State Academy of Arts, Stuttgart, died of a grave disease on March 15, 1948 at the age of 60. He was one of the group of leading German Paint Scientists including Professor Eibner, who died about 15 years ago, Dr. Hans Wolff, who died in 1944, and Prof. Wagner, many of whose papers were published in PAINT AND VARNISH PRODUCTION MANAGER. The Paper on MECHANISM OF PIGMENTATION and the present Paper were the last two treatices written by the deceased. We hope to be able to publish a few other of his posthumous papers in due time.

and monoclinic lead chromate. These pigments were added either alone or in various mixtures with one another.

The films containing red lead, red oxide of iron and zinc chromate (chiefly of granular structure) proved to be of typical granular surfaces and cross-sectional structure. The grains themselves represent aggolmerates of primary particles and appear to be glued together in strong, dense masses. The dry films do not exhibit larger pores and the interstices between the pigment agglomerates are filled with oil. The question arises whether these agglomerates are formed only during the drying period, i. e. whether the primary particles remained separate after wetting with the oil, agglomerating only within the dispersed system. In this case, they are thoroughly wetted throughout and are separated from each other by oil films even within the secondary agglomerates. It is true that in the presence of oleic acids these separating films may be of practically monomolecular thinness.

In order to solve this problem we mixed dispersed red lead into a wetting agent, ground the mixture together with oil on glass plate and subjected the film to micoscopic examination. We found that even in fresh mixtures in which the particles were still in movement, agglomerates could be discerned, although their dimensions could not compare with the agglomerates present in normal red lead paints. Further agglomerations were formed until the movement of the particles stopped entirely but the system then remained stable until perfect dryness was attained. It appears that at least a considerable portion of the pigment particles form secondary agglomerations even during the wetting treatment and are not therefore surrounded by oil films. Air inclusions are possible during the first stage of mixing, but it is probable that this air is removed during technical grinding-in of the mixtures without inducing wetting of the particle surfaces with oil. If this assumption is correct and if the agglomerates therefore act as primary particles whose total surface is smaller than the combined surfaces of all individual pigment particles it is obvious that a certain amount of the active pigment substance within the mixtures is useless as far as corrosion protection is concerned.

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Changes of shape and circumference of the secondary agglomerates take place during the drying process which tend to create "halo"-spaces around these formations probably due to dynamic processes. We have studied the character of these processes and connected with the red lead-linoleic acid reactions and have been studied and determined by photographic observations. We were unable to determine anisotropic lead soaps in any of the instances observed but were able to prove the reactions between red lead and oil with the aid of the hydrogen sulphide test developed by W. H. Droste.

The color photographs produced indicate the existence of "halos" of intensely brown coloration around the red lead particles which is not due to factisation of the oil.

The granular zinc chromate exhibits an arrangement similar to that of red lead within the paint films. We were unable to observe anisotropic soaps in polarized light, although this is no proof against this reaction which cannot be proven even by so clear a test as the formation of sulphide in red lead soaps. The film is exceedingly soft and easy cuttable. Monoclinic, acicular chromium yellow pigment particles do not arrange themselves along the brush marks as is frequently believed (and formerly believed even by ourselves) but in rather bulky aggregates, leaving comparatively large spaces free of pigment and filled with oil. It is for this reason that the film remains exceedingly soft and pliable and that it exhibits a long drying period. The pigment needles are blackened by the action of hydrogen sulphide but there are no brown zones indicating the formation of soaps, which is chemically obvious, of course.

Iron mica and aluminum powder, representing scaly pigments, exhibit the well known fish scale arrangement. The interstices are filled with oil, but we were also able to determine actually hollow spaces. The so-called "floating effect" can be eliminated by degreasing the aluminum powder. Iron mica with horizontal arrangement exhibits pronounced sedimentation within the films, inducing the formation of a top zone impoverished of pigment which is bound to deteriorate the protective capacity of the paint films.

If scaly and finely granular pigments are mixed, horizontal arrangement of the pigments and sedimentation phenomena are observed if the specific weights of the pigments are very different. In the case of a red lead-iron mica mixture the latter preferrably settles in the lower layers of the film parallel with the base surface; the central layers of the film contain most of the red-lead, while the top layers carry comparatively little pigment.

If the film produced on the specimen-holder of the microscope is observed from the lower side it is noticed, however, that the innermost layer of the film does not consist of mica scales but of a layer of red lead particles forming the adhesive

layer. In the case of iron mica-zinc yellow mixtures almost all of the latter pigment remains within the top layer. Observation of the film from beneath shows that zinc yellow also remains beneath the iron mica scales but that on account of a lesser tendency toward seggregation there is less of this pigment than of the corresponding red lead particles in the other mixtures. The mixtures of aluminum powder with heavy spar will be discussed later on.

B. Properties of the Film

1. Degree of penetrability (Porosity)

If well dried films are cut across, the cross section appears as though the oil-wetted agglomerates are but losely connected with each other and as if clearly recognizable hollow spaces are formed between them. However, if we prepare cross sections of films containing comparatively small quantities of pigment we find that these intervening spaces between the agglomerates are filled with the gel of the dried oil. We have been unable in any case to observe any faulty spots free of oil in any of the films containing granular pigments. If films of this type exhibit a high degree of penetrability towards liquids (moisture) this cannot be due to the presence of air in these faulty spots but must be caused by the swelling of the film substance.

The fact that paint films are penetrable not only to gases but also to fluids can easily be proven in the case of lead paints by the blackening reactions induced by hydrogen sulphide. Films of measured thickness and applied to glass plates were exposed to the action of a hydrogen sulphide current, hydrogen sulphide water and ammonium sulphide solution, the degree of penetrability of the films being measured by the length of time required to cause blackening of the inner surfaces of the films (adhering to the glass).

Linseed oil films 0.04 mm (40μ) thick are blackened throughout by ammonium sulphide within one hour. Blackening by hydrogen sulphide gas is effected within a shorter period, while hydrogen sulphide water acts less rapidly. In the case of ammonium sulphide the film is attacked by ammonia, of course. Better results were obtained with red lead-alkyde resin films which required about twice as long for thorough blackening.

It is not surprising in view of the high degree of volatility of ammonium sulphide that a high pentrability was determined in all tests employing this reagent, and it must be supposed that this action is more of a gaseous action in spite of the fluid character of the reagent.

In order to test the degree of penetrability of the paint films to fluids tin foils were primed with glutoline solution containing iron chloride, the test films applied and dried. The film was then placed into water for six days, followed by six days of intermittent watering and exposure to uviol light and finally spotted with a potassium ferrocyanide solution which was renewed for two days. The degree of penetration exhibited by the films tested was then indicated by the appearance and intensity of Prussian-blue formation on its surface. It was found that this reaction was not developed in the films pigmented with red lead, red lead-spar mixtures and red oxide of iron, while a few blue reactions were observed in the films pigmented with iron mica and with aluminum powder, indicating sufficient porosity at the spots involved to permit of direct contact between the fluid reagents and the base.

We therefore concluded that finely granular pigments tend to yield films exhibiting a much lower degree of penetrability to fluids under the testing conditions selected than the scaly pigments in absence of finely grained space-filling pigments.

We now attempted to determine the degree of porosity by electrographical methods using paint films applied to sheet iron strips which served as electrodes. A glass cylinder was placed on to the paint film and fastened with dough. This was filled with water in which we dipped the counter electrode. The distance between the film and the counter electrode was 20 millimeters; the electric tension amounted to 2 Volts. The results obtained

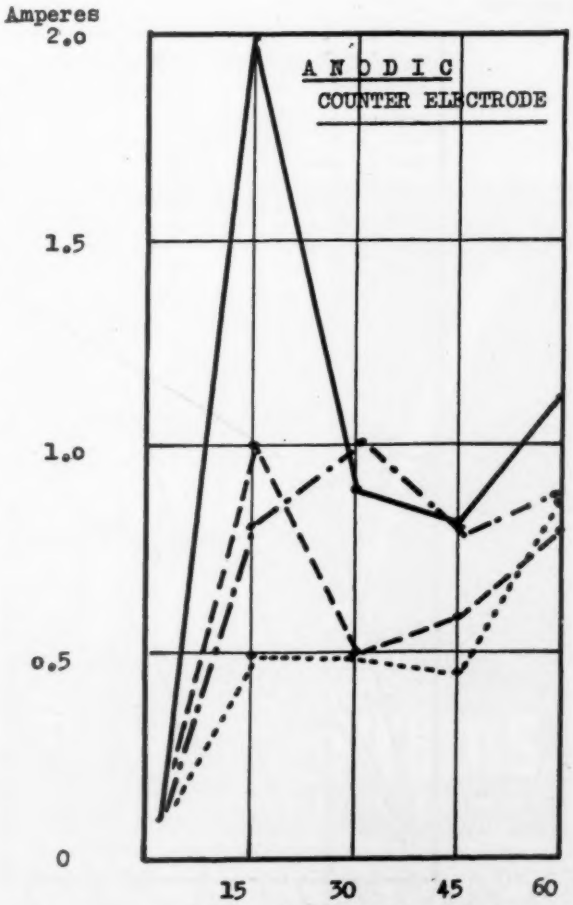

Diagram No. 1

are indicated in the two diagrams which show the voltages required for the various films within the periods of tests selected. The diagram No. 1 shows the results obtained with anodic counter electrodes, diagram No. 2 the results with cathodic counter electrodes.

The diagrams show that higher results are obtained with anodic counter electrodes, average maximum results being obtained with the aluminum bronze paints in both groups of tests. The question arose whether the values obtained are not considerably influenced by the electric conductivities of the various pigments. In order to test this possibility we conducted counter tests with the anodic counter electrodes in direct touch with the paint films. We obtained the following results:

red lead paints	1.8 Amp.
iron oxide paints	14.5 Amp.
aluminum paints	14.5 Amp.
red lead-spar paints	8 Amp.
iron mica paints	14.5 Amp.

It appears obvious from these results that the paints containing red oxide of iron, iron mica and aluminum powder exhibit a very high degree of electric conductivity, only the films containing red lead or red lead mixtures yielding certain conclusions as to the degrees of ionic penetrability.

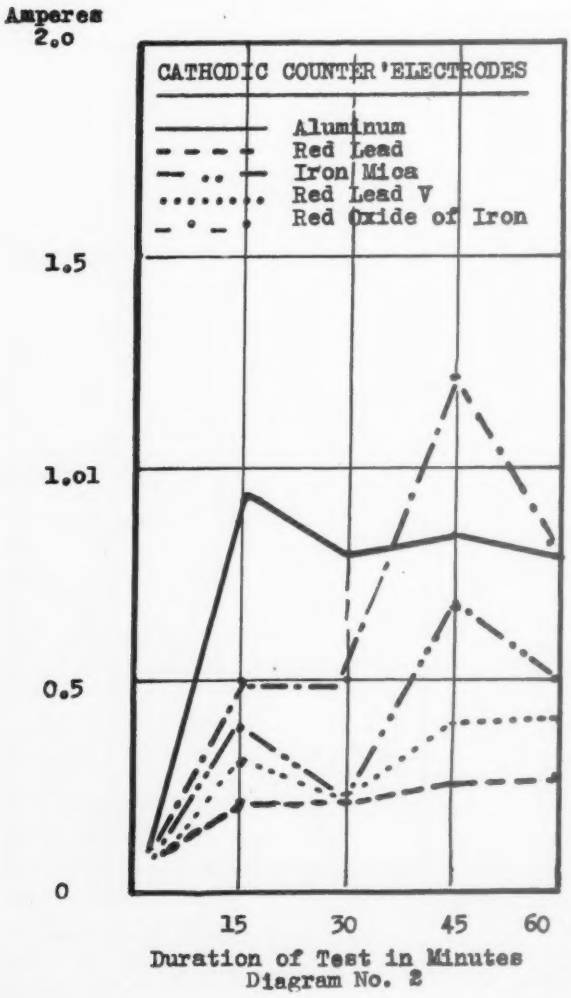

Diagram No. 2

In view of the fact that electrographic tests operating at high electric tensions are bound to yield unreliable results for the reasons indicated we had similar tests conducted by J. K. Wirth who applied his ionic penetration testing method operating with ordinary local currents. His report was submitted as follows:

Red Lead

The curves obtained were almost perfect and remained almost on the O-line during the entire testing period, indicating a very low degree of ionic penetration, although on the fourth or fifth day the film developed a very slight rust spot. Shortly after the appearance of this spot the galvanometer indicated a current increase and some fluctuations but the curve returned to the O-line soon afterwards. This "healing process" is not unusual in paint films exhibiting a satisfactory W_1 and W_2-curve.

Red Oxide of Iron

The results were less satisfactory. The J_0-curve remained near the O-line a number of days. It then commenced to rise, slowly at first but more rapidly later on.

The red lead-spar paints and aluminium paints indicated strong current penetration a few seconds after commencing the tests and the galvanometer showed the typical zigzag curves indicating a high degree of discontinuity of the films tested.

The results obtained by J. K. Wirth thus agreed rather closely with our own results, although the possibility exists that even in these tests the electric conductivity of the pigments influence the results obtained.

(2) Contact Between Pigment and Base Surface

We have also attempted to solve the problem whether contact between the pigment and the iron base is possible in a paint film of normal structure in which the pigment particles are surrounded by vehicle substance. For this purpose we prepared aluminum paint samples placed in weakly acidified copper sulfate solutions. In case of contact copper would have to be precipitated on the aluminum scales.

Under normal testing conditions copper did not precipitate. If, however, the smooth surface of the film was carefully cut away, copper was clearly precipitated on the aluminum scales after a short time, which proved the possibility of contact in presence of water and of an electrolyte (although there is a remote chance that by cutting away the film surface the oil films were partially damaged).

Nevertheless, it appears certain from these results that electro-chemical reactions between pigments and iron base surfaces are really possible and that the frequently doubted passivating influence of pigments really exists.

(3) Influence of Water

In order to determine the influence of water on the anti-corrosion properties of films we first prepared pigments of different water contents. The pigments employed were red oxide of iron and zinc chromate. (1) air-dry, (2) after heating to 250°F and (3) with 5% of water. The films were prepared by mixing these pigments with linseed oil, drying the films and immersing them in water for three months.

It was found that there was little difference between the films containing red oxide of iron used in the dry, semi-dry and moist condition. All these films had developed blisters containing rust. Much different conditions were observed in the zinc chromate films. The air-dried and dehydrated pigments induced the formation of large blisters containing considerable amounts of rust, while the films containing the pigment with 5% water developed only comparatively few small blisters, the iron surface beneath them remaining completely bright and unrusted. This proves that the presence of water in the zinc chromate particles (which are slightly soluble in water) induced the passivating influence of the pigment. It must be added, however, that this favorable influence of the moist pigment on the anti-corrosion properties of the films is accompanied by a deterioration of the mechanical properties of the films after three months of exposure to water. Shreds of film removed from the sample proved it to have become thoroughly brittle.

II. CONCLUSIONS

A. Improvement of Anti-Rusting Pigments

The basic experiments described above indicate that finely granular pigments of high quality yield satisfactory anti-rusting films but that on account of their tendency towards agglomeration within the films only the actual surfaces of the agglomerates remain active in this respect while the pigment surfaces within remain inactive. There can be no doubt, therefore, that under conditions favoring agglomeration there is a pronounced waste of pigment materials. The results also indicate that the theories concerning the favorable influence of the scaly pigments are theoretically justified but that in their case there is the danger of the formation of hollow spaces and porosities within the films. It is necessary for this reason to attempt to fill up these voids within the film structure.

(1) Addition of Fillers to Scaly Pigments

If aluminum powder is able to react with fatty acids leading to the formation of soaps it should be possible to obtain better results with this pigment than with iron mica powder, since the unpigmented interstices between the pigment aggregates can be filled up with lamellar soap products improving the elongation and resilience of the films. However, there is also the danger that in presence of free fatty acids hydrogen is formed inducing the formation of foamy sections within the films.

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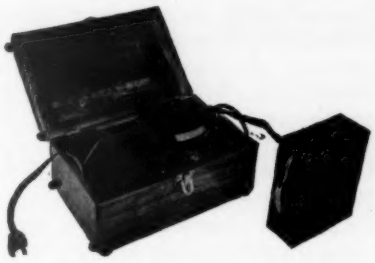

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Acting on this idea we first attempted to treat degreased aluminum powder with an oil of high acid number on one hand and with another oil in which the free fatty acids were neutralized by prolonged reaction with 10% of zinc white. In order to observe a possible formation of hydrogen gas paints of the various composition were not only applied in form of films but were also stored in tightly closed tins. We were not able in any instance to observe the formation of hydrogen, which agreed with the technical literature denying any reactions between aluminum and fatty acids. We wish to point out however, that this does not apply to aluminum oxide, and since all metallic aluminum is covered with a thin oxide film, there should be a certain formation of products of reaction even if the amounts of these products formed may be exceedingly small and without influence on the properties and structure of the films.

It therefore appears desirable in the case of aluminum paints to supply suitable inert fillers resistant to swelling. We studied mixtures of aluminum powder with 50 per cent of heavy spar, asbestine, slate powder, silicon carbide, zinc white and other mixtures containing 25 per cent each of zinc white and heavy spar as well as zinc white and slate powder. The films were applied to sheet iron strips. ⅓ of the test films were left uncovered, another ⅓ was provided with a white lead top coat colored gray and the last ⅓ with an iron mica top film. Part of these samples were freely exposed to the roof atmosphere of our laboratories while the others were submitted to salt spray tests and to moist air atmospheres in the laboratories of the METALLGESELLSCHAFT, Frankfurt on Main. These latter experiments yielded the following results:

Degrees of Rusting

	Salt spray tests (42 days)			Moist Air Chambers (57 days)		
	I	II	III	I	II	III
1) Aluminum-Spar	4	2	1	1	2	1
2) Al-Asbestine	2	1	2	2	2	2
3) Al-Slate powder	3–4	1	1	1	1–2	1
4) Al-Zinc white	2	2	2	1	3	1
5) Al-Spar-Zinc white	2	3	2	1	2	0
6) Al-Slate-Zn-white	3	2–3	2–3	1	1	1
7) Al-Silcar	5	3–4	2	1	2	0

I = freely exposed test film
II = test film covered with gray white lead film
III = test film covered with iron mica film

The primary character of these tests is indicated by the fact that the results of the salt spray tests and of the moisture chamber differed considerably. For instance, while the mixture No. 6 (Aluminum powder + slate powder + zinc white) yielded very satisfactory results in the moisture chamber, they proved very weak under the salt spray. The same applies to mixture No. 7, etc. If the salt spray tests are considered most reliable the asbestos mixture appears to be most satisfactory, while the slate powder-zinc white mixtures and the silicon carbide Mixture (No. 7) yield best results under the conditions of the moist air chambers.

Microscopic examination of cross sections of the various films subjected to these tests indicate that, as expected, the structure of the films is considerably influenced by the addition of the various fillers. These examinations also prove that—as already indicated in the above tables—the white lead top coats do not yield as satisfactory results as the iron mica films. Practical experience with anti-rusting films confirm this result. It is well known that the usual type of top coats are not satisfactory on priming coats containing scaly pigments.

(2) Production of "Plated" Pigments

In combining scaly pigments with fillers it may possibly be of considerable practical advantage to employ an active anti-corrosion reagent instead of the non-active fillers. These mixtures could possibly be effected so that the active fillers represent the characteristic anti-rusting substances while the scaly pigments would form the pigment basis. This would apply to lead compounds such as red lead, litharge, lead sulphate or even metallic lead powder—in connection with aluminum or iron mica powder. Mixtures of aluminum powder with lead pigments have always been avoided for electrochemical reasons, however.

This theory is justified if red lead actually exerts an electrochemical influence. The investigations of W. J. Mueller seem to prove, however, that the action of red lead is of a different nature, and that its use with well selected mixtures of scaly pigments would lead to satisfactory results. The trouble remains that in mixtures of this type the lead pigments would tend to agglomerate, reducing the total efficiency accordingly, while the value of the films as a whole would be lowered by sedimentation.

It is therefore logical to suppose that the structure of the pigment contents of a film could be arranged so that the filler material would form a skeleton structure covered entirely by an anti-corrosion substance. This idea is not new by any means. In his paper offered at the 74th General Meeting of the German Society of Engineers (VDI) at Darmstadt in May 1936 B. Scheifele first pointed out the advantages of "plated pigments" in which particles of inert pigment are surrounded by a film, or surface layer, of active anti-rusting pigment material such as red lead, etc. No satisfactory practical process for the production of such plated pigments has been developed as yet, the simple precipitation processes suggested and carried out during recent years appearing entirely unsuited for this purpose. We have carried out large series of experiments to develop useful practical processes for the production of plated pigments or, as we prefer to call them, "nucleus pigments". We were able to study four useful types of processes: (1) the purely chemical processes, (2) colloid-chemical methods, (3) structural-chemical processes and (4) electrochemical methods of production.

(a) Chemical Methods of Production

These are possible only with scaly pigments of metallic character and easily oxidized. A typical example of this type is degreased aluminum powder treated with sodium chlorate or ammonium persulfate or any other suitable oxidizing agent. A light gray pigment is obtained which is able to react strongly with fatty acids. This pigment consisting of a nucleus of metallic aluminum and a surface layer of loosely adhering oxide is called Oxal.

(b) Colloid-Chemical Processes

If lead salts are permitted to crystallize out on substratas such as heavy spar, slate meal, asbestine, etc. the product formed on the latter does not form a film or a connected surface layer. Films of that type can only be produced from solutions if the corrosion-active substance is able to form gels surrounding the nuclea in form of films. We worked on the theory that such films should be producable with organic non-crystallizing lead compounds soluble in hydrocarbons. During our search for such compounds we hit on a substance developed by Professor Dr. Ott, of the technical University of Stuttgart. This substance, tridodecyl lead,

$$Pb^{III}\ (C_{12}H_{25})_3$$

is employed as active oxygen carrier (Patent applied for on January 7, 1941). This substance is soluble in cold hydrocarbon solutions as well as in hot fatty oils. Our first series of tests were carried out with solutions of this substance in benzene. Heavy spar powder were treated with this solution and the benzene was distilled off. The "plated" pigment produced in this manner was immediately ground with oil. The film samples produced were subjected to open air weathering tests which are still continued at this writing.

(c) Structural-Chemical Methods

The ideal of a "plated" pigment has been furnished by nature in form of the well known Veronese Sienna which represents a form of dolomite rhombohedrons of zonar structure produced by fractionated mixed crystallisation. The interior black zone consisting of dolomite and iron carbonate is surrounded by a colorless zone consisting of pure dolomite. If it is possible to produce similar mixed crystallisation with lead salts and suitable filler substances, plated pigments can be produced in the same manner, provided that the crystals produced are of sufficiently small dimensions to prevent destruction of the aggregates during the grinding procedures with the paint vehicles.

Studying this problem from a practical point of view it appears probable that mixed crystallisation can be effected only with mixtures of barium and lead sulfates such as takes place in the manufacture of lead chromate. We have not yet been able to determine whether simultaneous precipitation of the two components produces only

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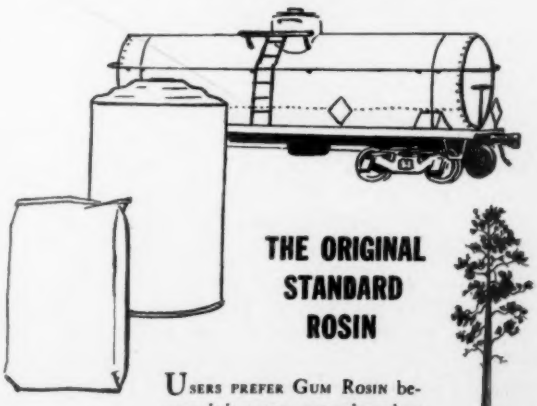

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a common mixture of particles or true mixed crystallisation. This problem can only be solved by röntgenographic methods.

We have also attempted to obtain satisfactory results by dissolving barium sulfate and lead sulfate in concentrated sulphuric acid and to obtain fractionated crystallisation by the addition of water to this solution. We have not carried out röntgenographic examinations as yet because the method outlined offers a number of practical difficulties, while the pigments produced thereby are of a rather spongy character exhibiting excessive oil requirement.

(d) Electrochemical Processes

If lead salts, representing the salts of a comparatively noble metal, are brought into contact with metallic powders such as aluminum, zinc and magnesium, the nobler metal (lead) is precipitated onto the "nucleus" in form of a continuous thin film. This process represents the simplest and most economical method of producing scaly plated pigments.

Degreased aluminum powder and an aluminum-silicon alloy (Sigal) were treated with a weakly acidified solution of lead nitrate or lead acetate respectively, the process being conducted in such a way as to get a precipitate of about 10% of lead on to the metal powders. The material is then dried, crushed and ground with oil or an oily vehicle (alkyde resin). A pigment produced in this manner is called Plumbal. It is of grayish appearance. If it is superficially oxidized by oxidizing agents in hydrous solutions, a somewhat lighter pigment is produced which we have termed Plumboxal.

Since the lead film on Plumbal is not exceedingly adherent, we tried to improve its adhesion by the well known galvanostegic process. Degreased aluminum powder was made up with water to form a paste and charged into a crucible with perforated bottom. The crucible was placed into a solution of lead salt. A carbon electrode was dipped into the aluminum paste, while the crucible was connected up as cathode with the electrode. The lead plating of the aluminum powder obtained in this manner proved to be very adherent. The product was termed Electrolyte-plumbal. The properties of this pigment will be described later on.

Attention should here be called to a very peculiar phenomenon observed on testing the oxide layers of Oxal and Plumboxal. The test was based on the red coloration obtained with alcoholic alizarine solution. We found that Oxal did not turn red in spite of the fact that theoretically it should react clearly, while the reaction was well recognizable in the case of Plumbal and Plumboxal. It is true that the red color obtained exhibits a blueish tint, and it is well possible that the color is not really due to the formation of an alizarine lacquer but to the violet coloration of alizarine by substances of basic character which, by the way, can also be produced by the action of calcium oxide. Another fact pointing against the formation of alizarine lacquer by the oxide layer of the Oxal is that this layer is of a crystalline character. The violet coloration obtained with Plumbal indicates, however, that this pigment is also covered superficially with a fine oxide skin.

(3) Swelling Resistant Films Around Pigments

In conducting our experiments we also consider it advisable to determine whether we could not obtain an improvement of the paint films by protecting the pigment particles against swelling by providing a swelling-resistant organic film. We attempted to obtain such pigments by treatment of heavy spar with a Vinoflex solution, evaporating the solvent and using the product directly as pigment together with a certain quantity of corrosion-protecting pigments amounting to about 10 percent. (Red lead, aluminum powder and zinc white). However on grinding these pigments with oil we found that the oil or thinners respectively caused superficial swelling inducing the formation of streaky masses and the production of uneven paint films. It was found, however, that these films appear to be quite weather resisting, at least during the first period of our present exposure tests, so that in spite of their appearance they may be useful for certain purposes not requiring excellent looks.

B Improvement of the General Structure of Films

(1) Non-swelling and Uviol-Protecting General Structure

We carried out experiments to obtain a non-swelling structure of the films retaining red lead as basic pigment in linseed oil-boiled oil and the customary top paint (80 parts of red oxide of iron, 20 parts of zinc white and coloring with lamp black) by providing suitable intermediate layers between priming and top coat. Materials used by us for this purpose were Vinoflex, chlorinated caoutchouc, benzyl cellulose together with 5% of Clophene each for softening purposes. We also employed Durophene-218 V in order to avoid or reduce, penetration of ultraviolet rays. Sixteen different systems of these mixtures were subjected to short and weathering tests in which the substances enumerated were applied as priming or top coats between two red lead coats and the final top coats or as intermediate layers between two red lead coats. In another series of tests red lead pigments in normal grinding mixtures and in coats of very thin films were applied, the vehicles employed including alkyde resins. We also tested the Vinoflex layers in unpigmented condition as well as pigmented with the special heavy spar "Bakola"-preparation.

All these systems were subjected to short and weathering tests for more than a year without showing signs of deterioration. The weathering tests are being continued. Interesting are the short test results obtained with hydrogen sulphide.

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These tests applied to the fresh films yielded appreciable penetrations only in four instances, particularly where Vinoflex was used as priming coat. The system: two red lead coats and one gray top coat thus indicates a comparatively high degree of penetrability under the conditions of test selected by us. The degree of blackening observed from the backside (film applied on glass) was 3. By inserting a Durophene film between the red lead film and top coat the degree of penetrability was reduced to 1-2. A certain amount of penetrability towards hydrogen sulphide was exhibited only by those systems in which one red lead priming film was covered by an unpigmented Vinoflex film and a gray top film. All other systems proved to be impenetrable to three hours of exposure to flowing hydrogen sulphide gas, and no coloration was observed on their inner surfaces.

However, very different conditions prevailed after eight days of immersion of the fresh film samples in water. All films then exhibited a certain degree of penetrability, the degrees of blackening of their inner surfaces fluctuating between 3 and 5 with the exception of those systems in which the Vinoflex films and the chlorinated caoutchouc films were interposed between the two red lead films, also those in which the pigmented Vinoflex was applied on to the red lead films and in those in which the red lead paints were ground-in with alkyd resin. Another striking observation was that the system in which the Vinoflex was used as priming coat the paint films were considerably improved by watering, the degree of blackening being considerably less pronounced afterwards. This fact seems to indicate that the red lead-oil films are improved by watering. These short tests results can be satisfactorily evaluated only after obtaining the results of the weathering tests.

(2) Reduction of Thickness or Red Lead Priming Coats

The red lead films applied in the tests described above were of extreme thinness (i. e. as thin as could be applied under practical operating conditions). We thereby worked on the theory that in case of contact between the pigment and the iron surface to be protected and if the red lead actually exerts an electrochemical—or passivating—influence, only the red lead present within the adhering layer of the film acts as active rust preventative, so that an increase of thickness of the red lead priming films beyond that point would really be unnecessary. While in the tests described this aim was attained by thinnest possible application of the paints we conducted further experiments for the purpose of attaining the same end by changing the compositions of the paint, i. e. (1) by improving the degree of brushability, or the yield, of the paint, (2) by replacing part of the oil with Sangajol, (3) by producing thixotropic water-in-oil emulsions. The composition of these various paints were selected as follows:

	Red lead	Oil	Sangajol	Water
Paint No. 1	100	30	—	—
" 2	100	25	5	—
" 2a	100	25	10	—
" 3	100	30	—	10
" 4	100	20	—	10
" 4a	100	20	5	10

After six months of short-weathering tests the largest part of the exposed surfaces still is unattacked, the only signs of corrosion being swelling phenomena commencing at the lower edges and causing wrinkling and loosening of the films at the points in question. The degree of deterioration from this source with the various types of films is as follows:

Paint No.		Degree of deterioration	Thickness of films (mm)
1	Normal application	1.5	0.050—0.090
1	Thinnest possible application	2.5	0.030—0.055
2	Thinnest possible application	3.5	0.030—0.055
2a	Thinnest possible application	2.5	0.045—0.090
3	Thinnest possible application	0.5	0.035—0.070
4	Thinnest possible application	1.5	0.045—0.090
4a	Thinnest possible application	2	0.040—0.090

The weathering tests are still continued, the values given above representing intermediate results only. The hydrogen sulphide tests (30 minutes exposure to flowing hydrogen sulphide gas) indicated penetrations throughout the entire film thicknesses in all films excepting Paint No. 1 of normal thickness of application, in which the sulphide had penetrated by about ¾ of the entire film thicknes.

A very significant result is the behaviour of the paint film No. 3, especially if this is compared with the degree of deterioration of the heavier paint film No. 1 containing the same percentage of oil. This is doubtlessly due to the addition of the 10 parts of water, i. e. to the formation of the formation of the water-in-oil emulsion, and the good results obtained with this material are the more apparent since the films are considerably thinner than those of the normally applied films No. 1. Another interesting factor proving the improvement of quality obtained by emulsifying the oil is the comparison between Paint film No. 4 with the film No. 1 of normal thickness. Paint film No. 4 containing only 20 parts of oil and 10 parts of water (in form of an emulsion) is absolutely equal to the film No. 1 containing 30 parts of oil.

Film No. 4a of the same general composition as film No. 4 but containing an addition of 5 parts of Sangajol proved to be less satisfactory than film No. 4.

Attention should also be given to the fact that "thinnest possible" applications of the priming coats did not yield the expected results, the degree of deterioration of these thin films exceeding considerably that of the thicker "normally applied" films of similar composition.

The results obtained so far are of preliminary importance only. All the short and weathering tests are still continued and while the preliminary results may not be absolutely conclusive they doubtlessly offer very interesting and instructive material.

NEW ASPECTS OF RUSTPROOFING*

By ARTHUR MINICH, Nuodex Products Company, Inc.

You men of the paint and varnish industry have coped with the problem of rust prevention as a part of your over-all involvement in protective coatings. Indeed, one of the major functions of the many different products which you have manufactured is to protect metallic surfaces against corrosion and particularly, iron and steel, against rusting. The compositions which you have created for this important application have been of considerable latitude in respect to their make-up. You have employed various vehicles such as raw linseed oil, synthetic vehicles, etc. and you also had a choice among a number of pigments for the opaque type of rustproofing coating.

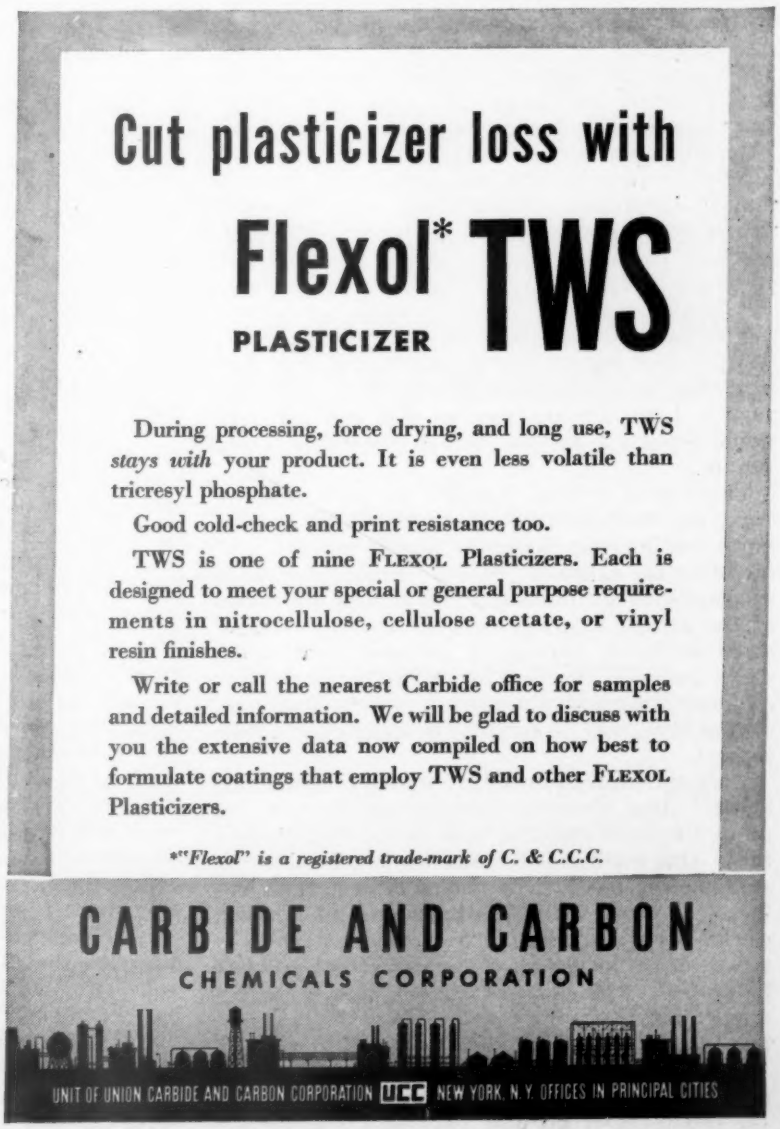
Small wonder then, that a great deal of literature has been published on the subject of corrosion control generally, and rustproofing specifically. To condense the great mass of evidence on this subject into a few minutes of discussion, we may say that rusting, chemically speaking, is the phenomenon involving the conversion of metallic iron into iron hydrates of the ferrous and ferric form. The primary reaction is that which takes place between iron and water. This in turn results in liberation of hydrogen which is attacked by oxygen and thus removed from the surface. The reaction requires the presence of (a) water or moisture, (b) oxygen, and (c) an acid. It has been generally proved that rusting will not take place in the absence of acid, even though the other factors may be present. If, for example, an iron surface is exposed to air and water, from which carbon dioxide has been entirely eliminated, no rusting will take place. Thus, acid is required to serve as actalyst for the reaction, and even as weak an acid as carbonic acid will suffice. Contrarily, rusting will not take place under alkaline conditions, even if the other factors, that is moisture or water and air, are abundantly present.

The generally accepted theory concerning the mechanism of rusting is the electrochemical interpretation. When a metal corrodes, it is oxidized to a higher valency by a loss of electrons, and some substance in the atmosphere or environment is reduced to a lower state of valency by the gain of electrons. When iron corrodes, it goes to the ferrous or ferric state with a loss of electrons, and the oxygen of moist air goes to the hydroxyl ion with a subsequent gain of electrons.

The total reaction precipitates ferrous hydroxide, which is gradually attacked by oxygen to form predominantly hydrated iron oxide.

It can be seen that this electrochemical mechan-

* Presented before the following Paint and Varnish Production Clubs: New York and Toronto, January, 1949; Chicago, February, 1949, and St. Louis, March, 1949.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ism of corrosion is closely related to a galvanic cell. Associated with the loss and gain of electrons is the flow of electrical currents between various parts of the metal surface at definite distances from one another. This is generally accepted since, in electrochemical corrosion, the resulting attack is confined to certain areas on the metal surface, while adjacent areas may be completely free from any signs of corrosion. This similarity is increased when one visualizes the metal areas as being the anode (the corroded area) and the cathode (the unoxidized area) with a difference of potential. It is this difference in potential that is responsible for corrosion. The amount of current flowing governs the rate of corrosion and consequently the amount. Such differences in potential may be due to a number of factors, that is, differences in oxygen and metal ion concentration. The actual rate of corrosion in any particular case is related to a number of variables. To list the important ones: electrode potential, meal ion concentration, second phases, temperatures, pH values, etc.

Whatever the theories concerning the mechanism of rusting may mean, one thing is certain—that rusting of iron represents one of the most serious of all wastes of manufactured goods. It has been estimated that the annual loss through corrosion represents several billion dollars. It has also been estimated that of all the iron and steel products produced annually, about five per cent or used for replacement of material rendered unserviceable because of corrosion. Another estimation of the rust damages arrives at the figure of two per cent annual loss per total iron and steel in service throughout the world. This total tonnage is 1,200,000,000 tons, so that two per cent would represent no less than 24,000,000 tons per annum.

There have evolved through the years broadly two types of protective systems to prevent or minimize rusting, one being the anodic process, and the other the cathodic process. Anodic control is accomplished by impeding the rate of corrosion at the anode, whereas cathodic control is associated with the increase of the total polarization at the cathode. Various protective coatings, such as chromates and phosphates, drying and non-drying films, may be assumed to accomplish anodic control.

Cathodic protection offers a means of protection against stray electrical currents and currents set up by the small galvanic cells on the metallic surface. One method is to raise the potential of the surrounding medium by means of a storage battery. A second method is by the use of a highly electro-negative metal such as zinc, attached by means of an insulated wire to the iron object to be protected. Both these methods find particular application to the protection of buried steel and iron pipes of all kinds.

Two main reasons are given for the anodic control of films. (a) They cause an increase in the resistance of the path between the anode and the cathode, and (b) they decrease the rate of diffusion of the reactants and products of the electrode reactions toward and away from the electrode surface, thereby increasing the concentration polarization to values that may be much higher than those which prevail in the absence of films. It may, therefore, be said that anodic control by films is proportional to the resistance of the film and hence proportional to anodic polarization.

You have been especially interested in those rustproofing treatments and compositions where fairly heavy and rigid coating films are applied to the iron surface. There is no doubt but that these rustproofing coatings, which you may term rustproofing primers, have performed outstandingly well. However, it is equally obvious that for many applications, it is impractical or impossible to obtain rust protection through such compositions. There are many situations where iron surfaces must be protected against rusting, yet without affecting or interfering with their mechanical functioning. For example, iron or steel screws, nut and bolts, bearings, etc. are very vulnerable to rusting, yet they cannot be painted over with a rustproofing composition which deposits on them rigid films because they then lose their normal mechanical properties. There is also the industrial problem of furnishing interim rust protection to all types of iron and steel goods, especially sheet steel. For reasons of economy, as well as with the view of facilitating eventual removal of the protective composition, the normal kinds of protective coatings are unsuited.

This field has been but slightly tapped by your industry, but as we all know, the oil and grease industry owes an appreciable share of their business to the rustproofing application for their products. These compositions have generally not been offered or spoken of as protective coatings. An oil company might be surprised if they were classified as a paint company, yet their line will include some more or less efficient protective coatings of the type referred to.

In the belief that your industry might wish to grant broader cognizance to the inclusion of these non-drying, oily "protective coatings" within your total interests in protective coatings, our laboratory devoted considerable time and effort to the study of these "protective coatings." As the first step in this program, comparisons were made of various special rustproofing oils being sold by the oil and grease industry. As the first phase of our study, we gave careful consideration to the employment of proper test methods. We wished to employ such a variety of test methods so as to simulate the different kinds of field conditions under which such coatings would be expected to operate satisfactorily. As you are well aware, there are at the present time but three test methods in use, which are more or less common.

At the risk of boring you, may I briefly describe these methods.

1. *Modified Salt-Spray Method:*

This method differs somewhat from the standard salt-spray method which is based solely on appearance of test panels which have been exposed for a definite length of time to salt-spray vapors. The improved method permits, in addition to the visual rating, a reproducible and accurate quantitative estimation of efficiency.

This method employs the standard salt-spray apparatus, but the time of exposure and the size of the specimens are varied. Specimens used in this test are ordinary strap iron, 3 by ¾ by 0.05 inch thick. The specimens are prepared by dipping in hot 6N hydrochloric acid and flushing with hot water. They are then washed in hot alcohol and acetone and dried in a vacuum desiccator. The specimens are then weighed in triplicate, and the total weight recorded. They are dipped into the test oils and allowed to drain 30 minutes. They are then suspended, by means of a glass rod, in the salt-spray cabinet, and exposed to a salt-spray formed by a 20 per cent solution of sodium chloride for four hours at 100 to 110 F. A set of blank specimens is included, as well as the various test oils. At the end of four hours, the salt-spray is turned off and the specimens are allowed to remain in the humidified atmosphere at 100 to 110 F. for an additional 16 hours. The specimens are removed, washed with hot toluol, hot water, hot alcohol and finally hot toluol again. During this washing precedure, care must be taken to avoid any loss of rust particles. If this occurs, or if the rust appears in tree-like formations, the specimens are discarded and the test repeated. The specimens are dried in the vacuum desiccator and in triplicate, again recording the total weight of the three specimens. In evaluating the efficiency of the oils, arbitrary calculations are used, assigning a rating of 0 efficiency to a blank uncoated specimen.

2. *The Hydrobromic Acid Test:*

This test is, in most respects, the same as paragraph F-3a (12) of Navy Specifications 52-C-18. The only deviation from the method is the test specimen used. This laboratory employed specimens of polished cold rolled steel, No. 20, 1 by 1 by 0.0375 inch thick. The specimens are cleaned with benzol or petroleum ether and then dipped in hot alcohol. They are allowed to dry and then submerged in a 0.10 per cent solution of hydrobromic acid for one minute. They are removed from the solution and immediately dipped into the oil solution of the compound under test for one minute. The specimens are then removed from the oil solution and exposed in the laboratory. After four hours, the specimens are examined for corrosion or discoloration.

3. *The Steel Ball Corrosion Test:*

(This is a slightly modified procedure of the test found in *A.S.T.M. Bulletin* No. 139, March, 1946, in the paper "The Comprehensive Laboratory Testing of Instrument Lubricants" by Barker, Alter, McKnight, McKlveen, and Hood.)

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A. Steel balls 0.5-inch in size are used as specimens. They are polished with a form of emery powder, washed with water, alcohol, and acetone in that order, and placed in a vacuum desiccator until ready for use. A specimen is then placed in a wide-mouth test tube, 7/8 inch inside diameter, and covered with five milliliters of test oil. The tubes are then suspended in a constant temperature water bath at 60 C. After one hour, five milliliters of distilled water is added and the mixture shaken and then allowed to separate. The tube is then returned to the bath and allowed to remain undisturbed except for periodic examinations. The test is continued until one-fourth of the ball is corroded or until a period of 500 hours has elapsed. The time of testing, condition of the oil and water, and the corrosion of the ball are recorded.

B. In our laboratory, this test was employed also using synthetic sea water but because of the severe conditions, the duration of the test was limited to 300 hours or 13 days.

Recognizing that the above discussed test procedures do not provide an adequate picture of the rustproofing effectiveness of compounds, and also with the desire of having the test methods more closely related to service conditions, we developed certain new test methods to complement the others referred to.
They are briefly as follows:

1. *Steel Wool Immersion Tests:*

A. Pieces of steel wool, approximately three inches square, are dipped into the various oil solutions and allowed to drain overnight. They are then placed into separate eight ounce wide-mouthed jars, half-filled with tap water, and loosely covered. The rate or degree of corrosion is determined by the rust areas on steel wool and also the intensity of the rust color in the water. Results are usually expressed in the extent of rusting and the number of days required for the failure to develop. The duration of the test was 430 hours or 18 days. Compounds which inhibit corrosion for at least 15 days were found to be excellent inhibitors.

B. The above test is repeated using synthetic sea water. The duration of this test was reduced to 170 hours or 7 days because of the severity encountered. In general, it was found that compounds which showed poor corrosion inhibition, failed in 24 hours.

The above test method is exemplified by the exhibits I have with me which furnish very dramatic comparisons, yet this method may be carried out conveniently in the field, even by non-technical men.

2. *Nail Corrosion Tests:*

A. Ten-penny flat head nails, 3½ inch long, are dipped in hot toluol, alcohol, and finally acetone, and dried in a vacuum desiccator until ready for use. Number 7 cork stoppers are prepared by drilling a hole, slightly larger than the dimen-

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sion of the nail through the center. The nails are then placed through the hole with the flat head on top and then dipped into the various oil solutions. The nails are then drained one minute and placed in a two ounce round narrow-mouthed bottle half-filled with tap water so that the cork fits the mouth of the bottle snugly. Corrosion is similarly rated in the manner of the steel wool test above. However, to offer a real practical test, the duration of this test was limited to 170 hours or 7 days. This test is very severe and compounds which are poor inhibitors begin to allow corrosion in a few hours. Compounds which inhibit corrosion for at least 7 days are generally found to be excellent corrosion inhibitors.

B. The same test is followed using synthetic sea water. The composition of the synthetic sea water conforms to most specifications and is as follows:

C. P. Mg $Cl_2 . 6H_2O$	11.0 grams
C. P. Anhydrous Ca Cl_2	1.2 grams
C. P. Anhydrous Na_2SO_4	4.0 grams
C. P. Na Cl	25.0 grams

Distilled water to make one liter.

This method was also easily carried out and may be performed by non-technical men. Strangely though, this method was found by us to be the most severe of all methods checked by us and more oils and compositions which we investigated failed in this test than in all the other tests. A composition may perform very efficiently in the salt-spray sets, for example, and yet fail completely in this deceivingly simple test. To study the cause of this surprising phenomenon, we employed dyestuffs with the clear oils and we learned that the immersion of the nail into the water seemed to result in a stripping of the oil away from the nail. Even when excellent wetting agents were employed with the oils, this displacement of oil by water took place. This test method, we believe, is not only very interesting, but may also be very worthwhile from the standpoint of simulating conditions that may take place in practice. For example, when garden equipment and farmer's equipment is used, the exposed metal surfaces may be subject to direct water contact at times, such as may be caused by exposure to rain. The exhibit which I have with me illustrates that the test results seem to be indicative of actual exposure of the iron surfaces to rain.

After we arrived at the selection of the various test techniques discussed, we appraised several oils, including so-called rustproofing oils, and we learned that the straight mineral oils, while of some protective value in some of the tests furnished no protection in other tests. Likewise, the oils that are sold at a premium at rustproofing oils may offer improvements at varying degrees over the straight oils. It is no surprise, of course, that the various commercially available rustproofing oils necessitate their removal from the temporarily protected iron and steel surfaces when it is desired to ultimately apply the conventional types of protective coatings to them.

With the various test methods as our tools, we set as our next objective the development of a highly effective and concentrated rustproofing agent, which when added in small ratios to mineral oils or to volatile solvents, would provide protective coating compositions of high rustproofing efficiency. These efforts culminated in the development of a compound known as Nuodex Compound X-545. It represents, chemically speaking, an organometallic composition, embodying several active constituents. It was found impossible to obtain a high degree of performance in all tests through the use of a single compound. It was necessary to create a combination of several chemical agents.

Compound X-545 is in the form of a liquid and easily dissolved in mineral oils by simple mixing in the cold. (The compound also dissolves readily in volatile solvents, such as mineral spirits.) Dilutions of the compound to as low as 1.0 per cent transformed an ordinary mineral oil into a rustproofing coating material of good performance. By way of illustration, let me now furnish you with comparisons of a straight mineral oil, and the same oil modified through the addition low percentages of Compound X-545 into a highly effective rustproofing coating material.

1. *Modified Salt-Spray Exposure:*

	Efficiency Rating, per cent
Control Oil	36.0
1 per cent X-545	46.4
2 per cent X-545	78.3
4 per cent X-545	95.3

2. *Hydrobromic Acid Test:*

As little as 1 per cent X-545 added to the oil effectively neutralized the acid so that no corrosion was evident after the four-hour exposure period.

3. *Steel Ball Corrosion Test:*

Control Oil	*Tap Water*	*Sea Water*
	Fails—24 hr.	Fails—24 hr.
1% X-545	No corrosion—500 hr.	Slight—300 hr.
2% X-545	No corrosion—500 hr.	Slight—300 hr.
4% X-545	No corrosion—500 hr.	Very slight—300 hr.

4. *Steel Wool Immersion:*

Control Oil	*Tap Water*	*Sea Water*
	Fails—24 hr.	Fails—24 hr.
1% X-545	No corrosion—430 hr.	Trace—170 hr.
2% X-545	No corrosion—430 hr.	Trace—170 hr.

5. *Nail Corrosion Test:*

Control Oil	*Tap Water*	*Sea Water*
	Fails—24 hr.	Fails—24 hr.
1% X-545	No corrosion—170 hr.	Fails—120 hr.
2% X-545	No corrosion—170 hr.	Trace—170 hr.
4% X-545	No corrosion—170 hr.	No corrosion—170 hr.

In addition to the enumerated data, we also have information concerning the rustproofing efficiency of volatile solvent dilutions of Compound X-545. The test results to date indicate that even when this compound is applied in low concentrations—2 to 5 per cent in volatile solvents, e.g. mineral spirits—the deposited film furnishes very good rustproofing protection to the iron or

steel surfaces. However, on a quantitative basis the non-volatile oil dilutions are to be preferred.

The great advantage of the solvent dilutions of X-545 over the oil dilutions consists in permitting the subsequent application of conventional protective coatings without first removing the rustproofing film. We have established that when X-545 is incorporated into a non-volatile mineral oil it no more permits the direct coating of such surfaces with primers or top coats than would the unmodified oil. When X-545 is employed in the form of a volatile solvent dilution, this picture is different. It is then possible to apply regular metal primer and/or top coats without apparently interfering with the properties of these coatings. Whether this would hold true with all or even with the majority of protective coatings, we do not know yet—our tests have not been broad enough to permit a definite commitment on this point.

We believe that this completely balanced performance of rustproofing protective coating containing Compound X-545 as the additive, is unique and valuable. We believe that there exists a real need for such protective coating materials.

We feel enthusiastic about "our baby" for even with our jaded appreciation for technical developments, we cannot help but feel an addition agent, which in very low ratio and at relatively small additional cost, can transform an ordinary mineral oil into a protective coating material, is a step in the right direction.

I have attempted to convey to you the results of our thinking and work in the field of rustproofing. I hope that our efforts will prove to be of genuine service to your industry.

THE BLOOMING OF VARNISH FILMS*

By W. ESMOND WORNUM, M.C., B.Sc., A.R.C.S., F.R.I.C.

(*Continued from June issue*)

Now, as polar groups have a natural strong affinity for water, it follows that if, through adsorption of water, the secondary valences of these groups can be satisfied, swelling, with water as the liquid phase, will result as soon as opportunity and conditions permit.

Such swelling with water may initially take place in a purely additive way, i.e. water may be adsorbed in addition to any non-aqueous phase already associated with the system and without disturbance of the material. If the polar attraction is sufficiently strong, however, the non-aqueous "combined" liquid may gradually become displaced by the water. This would give rise to an oil syneresis, producing, as just previously suggested, what might be termed as *Syneresis Bloom.*

Instances clearly illustrating the swelling of varnish films with water, not only in the liquid state but also in the form of vapour, are not uncommon. Such illustrations are sometimes particularly marked in the case of certain alkyds, the clear films of which may be seen to rivel or wrinkle badly when brought into contact with water or water vapour. Such illustrations serve to emphasise that swelling with water can be a potent factor in bringing about physical changes in varnish films, even if the result of such swelling is not always so strikingly manifest as these illustrations might suggest. Nevertheless, where this wrinkling does arise through such swelling and is of microscopic dimension, it has the full character of a bloom, and this defect might well be referred to as a *Wrinkle Bloom* to distinguish it from other types of bloom.

Now it must be appreciated that, in swelling, the vapour pressure of a "combined" volatile liquid differs from that of the "free' 'liquid. As swelling is itself a differential phenomenon, in that the forces operating in the early stages of swelling are far greater than those after appreciable swelling has occurred, the vapour pressure of a "combined" liquid cannot be constant but will vary with the degree of swelling; it will also be a function of the secondary valence activity which is its primary cause. This is illustrated, in respect of water, in the case of the jelly-fish, which in its dehydrated form weighs less than one per cent. of its original weight but yet can remain in the sun without being dried up. If the vapour pressure of the "combined" water equalled that of the "free" liquid, this could never happen.

The importance of this vapour pressure consideration lies in the fact that whenever the vapour pressure of the "combined" liquid is less than that of the "free" vapour in the surounding air, continued swelling will result, and vice-versa. This means that water will be taken up by a varnish film, through the agency of swelling, whenever the relative humidity is higher than the vapour pressure of the "combined" water in the surface of the film. The stronger the polar activity, the greater will this water adsorption be and the lower the relative humidity at which swelling will take place. Different varnishes will thus be expected to behave differently in regard to the relative humidity at which they become affected and the amount of swelling which occurs under the same conditions of exposure. This is fully in accord with the views on surface polarity put forward by Morrell.

It follows from these considerations that, in the case of strongly surface-active varnish films, in which there has occurred an appreciable amount of non-aqueous swelling either during heat treatment or on drying, Syneresis Bloom can be induced at humidities below the saturation point. In other words, actual condensation of moisture on the surface is not a pre-requisite of such bloom formation. This is a very interesting point be-

cause undoubtedly blooming does occur under conditions when there is no visible manifestation of any actual condensation on the surface. In fact, in one series of exposures of blooming and non-blooming varnishes under a controlled humidity of 90 per cent., when actual condensation did take place, it was found after one week that none of the blooming varnishes had bloomed but all the non-blooming varnishes had whitened and were most unsightly.

That bloom formation is accompanied by an adsorption of water has already been recorded by Morrell. When the water adsorptions of blooming and non-blooming varnishes on immersion in water are compared, however, it is found that non-blooming varnishes do actually absorb more water than blooming varnishes. Morrell, for instance, gives the following figures:—

Carriage varnish, non-blooming, on immersion in N/400 NaCl, 3 days, 13 to 17 per cent.; 7 days, 22 to 25 per cent.; 14 days, 33 to 36 per cent.

Boat varnish (tung oil-rosin ester), blooming, same immersion, 3 days, 3 to 6 per cent.; 7 day, 7 to 11 per cent.; 14 days, 10 to 18 per cent.

In view of the discussion so far, and the influence which water adsorption has been seen to have on bloom formation, these results need some explanation. It is submitted, then, that these differences can be explained by reference to the difference in the character of the adsorption in the two types of film. Thus, in the case of blooming varnishes, such as the Boat Varnish, the adsorption takes the form of swelling, whereby any surface capillaries are closed and free passage of water into the film is prevented. In non-blooming varnishes of the Carriage Varnish type, as indicated by the contact angle, the polar activity is of a low order and very little, if any, swelling arises. The passage of water into the film is therefore mainly mechanical, as evidenced by whitening. In the course of time a distension of the surface structure and a re-orientation of polar substances in contact with the water are prone to occur, leading to permanent damage to the film and a chalking of the surface on drying out.

The precise conditions under which Syneresis or Wrinkle Bloom is most prone to occur are as yet undetermined. As these types of bloom involve swelling as a primary cause, it may be said that this would be intensified at lower temperatures, owing to increase secondary valence activity, and would diminish at higher temperatures. As films will always tend to reach a state of equilibrium with the surrounding atmosphere, it is probable that rapid changes in humidity and temperature cause greater disturbances in the surface of a film than prolonged exposure to a fairly constant atmospheric condition. Thus a changing condition, particularly when a fall in temperature is in-

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volved, is a positive factor in accelerating bloom formation.

The temperature and humidity at the time of setting and drying of the film will also exert a definite influence, because the prevailing conditions will largely determine the ultimate film structure, the orientation of polar groups, and the amount of swelling taking place through adsorption of non-aqueous liquids at the time of gelation and drying. All these are indeterminate factors which are very different to analyse.

Nevertheless, it is hoped that the physical and colloidal considerations presented in this paper will serve as a further contribution to the study of some of these various aspects of bloom formation. While it is appreciated that adsorption of water, by swelling, has been attested a major factor in producing these physical changes, it is at the same time realised that this adsorption may also promote chemical changes, which yet remain to be studied.

Morrell, for instance, has many times emphasised the importance of peroxide groups as a factor in bloom formation and, whereas these may well function as polar groups in relation to water swelling, causing physical changes, they may equally be centres of chemical breakdown under the agency of the adsorbed water. Aqueous swelling may promote other forms of hydrolysis, leading directly to chemical degradation of the surface of the film, producing all the characteristics of a bloom.

A recent chemical approach to the study of blooming, from a new angle, has been commenced by Morrell and Phillips[3] in their investigation of the action of varnish films on photographic plates. They have found that blooming varnishes cause a fogging of these plates and on their evidence they put forward the view that the active peroxide grouping is the cause of such fogging and that this is due to electron escape during polymerisation. They suggest that the escaping electrons condense water on the surface of the film, causing blooming. By studying the active peroxide content of varnishes oxidised with different catalysts, they further state different film structures are produced by cobalt on the one hand and by cerium or lead on the other; the longer the peroxide chain the less is the activity of the terminal peroxides, so that films containing cobalt are more active than those containing cerium or lead. The type of blooming here studied is that of Crystalline Bloom, with which Adam and Morrell were chiefly concerned in their study of contact angles. This work so far is of a preliminary nature owing to the war, but further results will be awaited with interest.

Enough has now been said to illustrate that the study of blooming, in its broadest sense, is one of considerable importance, not only in relation to the study of bloom formation itself but in the advancement it can make to the knowledge and wider study of the chemical and physical character of varnish films generally, and there is yet a great deal to be done in this direction.

In conclusion, it must be pointed out that, in order to simplify the investigations referred to in this paper and to aid its presentation, only the two extremes of varnish types, blooming and non-blooming, have been particularly studied and discussed here. It will be appreciated that between these types there is a wide range of varnishes, the properties of which approach one extreme or the other. In passing from one extreme to the other the behaviour of these varnishes can be logically correlated with their chemical and physical properties. The type of bloom, if any, most likely to occur in the case of any given varnish is largely determined by its previous history. The influence of materials controlling the building up of colloidal complexes, the action of driers and the consideration of other factors affecting film structure have been discussed in an earlier paper[4] and so have not been dealt with here.

3. R. S. Morrell and E. O. Phillips, *F.O.C.C.A.*, 1940, **23**, 233.
4. W. E. Wornum, *F.O.C.C.A.*, 1934, **17**, 119.

A NEW HORIZON FOR THE TECHNICAL MAN—MARKETING RESEARCH

By HAROLD M. JOHNSON, Vice-President In Charge of Sales, Nuodex Products Company, Inc., Elizabeth, N. J.

It is rather surprising that many words in common usage often mean one thing else to another. Therefore, I will take a few moments to make known my interpretation of the meaning of certain words in common usage which we will use in this discussion, thereby eliminating all possibility that I may be thinking one thing, and you another.

Let's take the key words in the title, and see what Webster has to say—here are his definitions, which, by the way, will explain to you my own usage of those words:

New, "Recently produced by change";

Horizon, "The limit of one's experience, knowledge or observation";

Technical Man, "Any one proficient or expert in technical detail, usually one versed in the technicalities of some subject, trade, profession or art";

* This paper was presented before numerous Paint and Varnish Production Clubs for publication in the *Official Digest* of the Federation of Paint and Varnish Production Clubs and is published by their permission.

Marketing, "The art or the business of buying or selling";

Research, "Diligent inquiry or examination in seeking or continued search after truth."

Technical men in the paint and varnish industry have many responsibilities:

They set up specifications and develop methods of testing raw materials; they develop processes for mixing and/or reacting these raw materials; they develop and adapt equipment for use in these processes; they develop specifications and develop methods of testing for the control of their manufactured products; they keep in touch with suppliers of materials and equipment through literature, attendance at meeting and personal contacts, so, that their firms may keep up-to-date with new developments; they formulate—they develop formulations for new products—they reformulate to improve old products—they reformulate to reduce costs—and they formulate to match competitive materials; they visit customers when there are complaints, and on occasion, they give product stories to their own salesmen and to their advertising and sales promotion department.

There can be no doubt in an honest-thinking person's mind but that the technical man has played a most important part in making the paint and varnish industry one of the most progressive of all industries.

So much for the technical man—while he is all puffed up with justifiable pride, let's leave him for the moment and talk about the SALESMAN.

A Salesman is that person in your organization who, having qualified prospects* for your Product makes personal contact with those qualified prospects and attempts to *create* and then *keep alive* in that prospect's mind a *desire:*

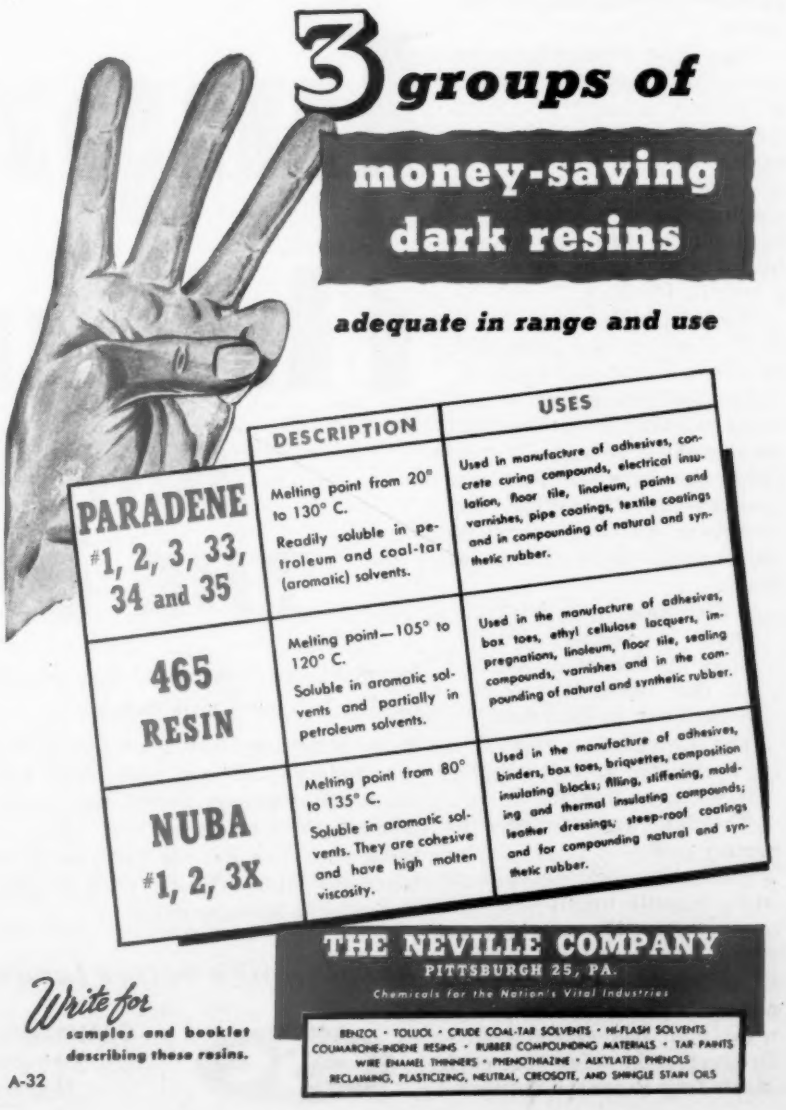
1. For your PRODUCT
2. To do business with your firm
3. To do business with him
1. That the prospect has a need for the product.
2. That he has the means to pay for the product.

*Qualifying prospects is the act of establishing:

A business, which has run-of-the-mill products, and depends alone upon a salesman-buyer relationship that is purely personal, is not a sound business. But if that salesman-buyer relationship exists, and the product is competitive from a quality and price standpoint, a sound business relationship will then exist as long as all factors remain the same.

And now, technical men, here is something that you should not forget—There are many top-notch salesmen who create desires to do business purely on a personal basis, but their activities will have no permanent benefit to your company unless you give them Products that are competitive in quality and price.

Now, let's look at that salesman who *creates* a *desire* in a prospect's mind purely on a basis of institutional selling; that is, he sells the firm. A very sound business relationship would exist under


3 groups of
money-saving dark resins
adequate in range and use
DESCRIPTION
USES
PARADENE #1, 2, 3, 33, 34 and 35
Melting point from 20° to 130° C. Readily soluble in petroleum and coal-tar (aromatic) solvents.
Used in manufacture of adhesives, concrete curing compounds, electrical insulation, floor tile, linoleum, paints and varnishes, pipe coatings, textile coatings and in compounding of natural and synthetic rubber.
465 RESIN
Melting point—105° to 120° C. Soluble in aromatic solvents and partially in petroleum solvents.
Used in the manufacture of adhesives, box toes, ethyl cellulose lacquers, impregnations, linoleum, floor tile, sealing compounds, varnishes and in the compounding of natural and synthetic rubber.
NUBA #1, 2, 3X
Melting point from 80° to 135° C. Soluble in aromatic solvents. They are cohesive and have high molten viscosity.
Used in the manufacture of adhesives, binders, box toes, briquettes, composition insulating blocks; filling, stiffening, molding and thermal insulating compounds; leather dressings; steep-roof coatings and for compounding natural and synthetic rubber.
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Chemicals for the Nation's Vital Industries
BENZOL • TOLUOL • CRUDE COAL-TAR SOLVENTS • HI-FLASH SOLVENTS
COUMARONE-INDENE RESINS • RUBBER COMPOUNDING MATERIALS • TAR PAINTS
WIRE ENAMEL THINNERS • PHENOTHIAZINE • ALKYLATED PHENOLS
RECLAIMING, PLASTICIZING, NEUTRAL, CREOSOTE, AND SHINGLE STAIN OILS
Write for samples and booklet describing these resins.
A-32

these conditions, too—but only if, and as long as the Product is competitive in quality and price.

Finally, there is the salesman who does an excellent job of creating a desire, in the buyer's mind, when he has a Product that is different than competitors', and if the difference *benefits* the buyer *in some way*. Please note that I use the word "different" rather than "better."

I hope that you have noticed the importance of the Product.

Summing up—there is *no* type of *salesman* who can be effective *permanently* unless the Product is Right!

Earlier, we made the statement that the technical man was responsible for the development of products—the technical man in the paint and varnish industry does have the "know how" as far as formulation and manufacturing are concerned, but the technical man cannot be assured that the Product he develops is commercially Right unless the fact that it is Right has been proved by Marketing Research.

Marketing Research, as an operation, may be defined, with strict adherence to Webster, as a "Diligent inquiry or examination in seeking facts or principles—laborious or continued search after truth" to improve the art of the business of buying or selling a specific Product.

Marketing Research, properly planned and executed, results in *Facts*—not opinions.

I recently heard of a conversation that took place that very clearly brings out the difference between fact and opinion, as Marketing Researchers, in their complete objectivity, must think of them:

> Two men were driving along a country road when one man said, "Look at the beautiful white horse over in that field." The second man replied, "I only see a horse that is white on this side."

The first was opinion—the second fact.

Marketing Research, as such, is quite broad and concerns itself with many matters which I feel should be handled by salesmen, statisticians and economists—once the technical man has given them a Product. I refer to such Marketing Research activities as qualifying prospects by area, by industry, by account; measurement of potential; appraisal of the effectiveness of marketing techniques; the determination of sales territories, and the establishment of quotas for individual salesmen, for area, for industry.

On the other hand, I feel that the technical man should play a very definite and important part in the collection and analyses of facts pertaining to the Product.

The technical man, alone, knows the limitations of formulation—he, alone, knows the limitations of his manufacturing equipment.

I am fully aware of the fact that most technical men today are performing certain Marketing Research functions, but in sort of a "hit-or-miss" manner.

Marketing Research, as a department of a business, is new, but any one who has been close to the operation of a Marketing Research function realizes full well that intelligently planned and in-

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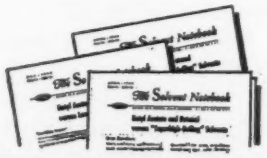

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telligently executed Marketing Research is going to be the best insurance that a business can have to continue healthy through the competitive era that we are just entering.

I sincerely hope that some of the technical men here have become sufficiently interested in this subject of Marketing Research to want to do something along these lines for your own as well as your company's benefit.

I will presume that this is a fact that some technical men here would like my thinking as to how they might get their feet wet in Marketing Research.

If I were a technical man, employed as you are by a paint and varnish manufacturer, I would do some reading on the subject of Marketing Research, or, as it is sometimes called, Market Research—to familiarize myself with all of the aspects of the operation, even though I planned to concentrate only on those aspects pertaining to the Product.

Most of the up-to-date information is found in Government and business association booklets and pamphlets, as well as trade papers devoted to the profession of selling, such as, Sales Management, Industrial Marketing, Printer's Ink, etc. I will be very happy to send anyone interested a bibliography on the subject.

After I had become, in a general sort of way, familiar with the operation of Marketing Research, I would have a chat with my Sales Manager. I would ask him to select two or three standard products which he felt could be sold in larger volume to active accounts, or which would be easier to sell to new accounts if they had certain "plus" features. I would ask him to define very clearly what "plus" features were desired, and ask him to procure samples of competitive products which he felt had those "plus" features that his standard products needed.

After I had heard his story, I would ask his permission to talk to a few of the salesmen and a few of the dealers to confirm what he had told me—to take the information out of the category of Opinion and put it in the category of *Fact*.

Whenever I had an opportunity of talking with customers, and our own salesmen and dealers, I would always try to get their thinking as to the weak points of our standard products, and conversely, I would also try to get their opinions as to the strong points of our standard products.

I would also ask my Sales Manager and my salesmen, whenever I had the opportunity, their opinions as to what new products, not now being produced, could be sold to active accounts—if we had them.

I would boil down all of the opinions and facts that I gathered from these various conversations and then select one of the recommendations and go to work and develop the Product that all of the people felt would be desirable.

In making my selection of the Product to be developed, I would bear in mind the availability of the raw materials needed, facilities for manufacturing, and profit potential.

After I had developed a product that met the exact specifications that those people had set up, and after I had compared it with like products of competitors, and I was convinced that I had the Product, I would prepare a presentation which would include exhibits of the Product I had just developed, and competitive products, proving all of the "plus" points by visual evidence.

When this presentation is ready, I would take it to my Sales Manager and to my Advertising Manager, to three or four of our star salesmen, each one being selected because he represents one of the types of salesmen mentioned before.

If the information I gathered in my preliminary discussions was *Fact*, not opinion, and if the Product I developed met the requirements based on Fact, and if my presentation for my own sales and advertising department was properly gotten up, there would be two worthwhile results:

1. A surefire PRODUCT that all types of salesmen could sell.

2. A sincere appreciation, by members of the Sales and Advertising Department, of the fact that the technical man had far greater potential in a business than the mere use of technical "know how" in the development and manufacture of products—the sales-minded individuals would be very grateful for "salesmindedness" in its technical men.

I am very grateful for the opportunity that you have given me to share my thinking on this subject with you—the talk has been a very short one—there are many angles that were not even hinted at, but I felt that all that would be necessary to stimulate your thinking would be a few Facts condensed, and that you, too, would see as I see—

Marketing Research as a New Horizon for the Technical Man.

FOREIGN PATENT

COATING SURFACES WITH POLYTHENE. Imperial Chemical Industries, Ltd. French Pat. 918,489. Applied for Dec. 7, 1945 (applied for in Great Britain Sept. 1, 1944); delivre Oct. 28, 1946. The adhesion of polythene to metallic surfaces is achieved by first coating the metal with a thin layer of a molten mixture containing at least 40% of a polyalkylene sulfide (e. g. the products sold under the trade names "Thiokol", "Novoplas" or "Perduren"). In practice, a mixture of a polyalkylene sulfide and polythene is melted, applied to a metal surface by a Schori pistol, and after cooling, a layer of polythene is applied similarly.

RECENT ADVANCES IN PAINT TECHNOLOGY

CHROMATED PROTEIN FILMS FOR THE PROTECTION OF METALS. A process has been developed for protecting steel, zinc, aluminum, and brass from corrosion by the use of chromated protein films. Either casein, albumin, or gelatin is applied to the metal surface by dipping. The film is impregnated with chromate, which acts as an inhibitor of corrosion and hardens the film, by adding the salt to the protein solution or by a separate immersion. These films may be useful for the protection of certain metals in mildly corrosive atmospheres. The chromated protein coatings represent a cheap, expeditious means of protecting metals from corrosion during indoor exposure, for example, during storage. They are superior to direct phosphate or chromate coatings or to oil films for this purpose, but their life is less than that of suitable paint coatings. The protein coatings are more effective on fairly large, regular surfaces than on the sharp edges and corners of small objects where the film tends to pull away. The protein solutions, unlike those used for direct phosphate or chromate treatments, do not become exhausted and require replenishment. After addition of the chromate, the protein solutions are fairly stable for long periods. The same solutions and procedures can be used for several metals. Metal surfaces must be entirely clean to ensure adhesion of the films. Plated articles may be taken from the plating bath, rinsed and coated without intermediate drying. The protein films can be removed by treatment with warm sodium carbonate solution, a more convenient and less hazardous procedure than the removal of oil films with solvents. A. Brenner, G. Ridell and R. Seegmiller. J. Electrochem. Soc. **93**, 55-62 (1948).

PIGMENTED RESIN EMULSIONS FOR PRINTING AND PAD DYEING. A short discussion of the properties, constituents and methods of formulating water-in-lacquer and lacquer-in-water emulsions is given. M. Silverman. Textile World **98**, No. 3, 230 (March, 1948).

SPECTROPHOTOMETRIC STUDIES OF THE OXIDATION OF LIONLEATES. Addition of carotene increased the oxygen uptake of ethyl linoleate, the initial rate approaching 2 instead of 1 mole of oxygen per mole of ester. Oxidation of linoleic esters by soybean lipoxidase was similar to autoxidation and from this the author suggests that the primary oxidation product is the same in the three systems studied. The absorption band at 2300-2350 Angstroms is due to the induced conjugated diene system, not to the peroxide group itself. The band at 2700-2800 Angstroms is due to enolizable substances, probably conjugated ketones. R. T. Holman. Biol. Antioxidants, Trans. 1st Conf. **1946**, 37-41 (Abstr. through Chem. Abs, **42**, 1616 (1948).

PROTECTIVE COVERINGS FOR MARINE APPLICATIONS. The present position of paints for ships' hulls and marine structures is reviewed under the following topics: effects of marine fouling, plant fouling, anti-fouling studies, effect of toxins, recent research results, uses of asphalts, bituminous paints, protection of internal surfaces, Admiralty corrosion committee tests, uses of plastics and flame spraying applications. Twelve references. R. Hammond. Paint Manuf. **18** 79-83 (1948).

FIRE-RETARDANT PAINTS. The Admiralty Chemical Department at Portsmouth is undertaking an investigation of interior fire-retardant paints, especially for ships. The discussion covers the factors involved in combustion of paints, the importance of primers, the formulation of top coats, experimental evaluation techniques, an inorganic cementiferous paint consisting of zinc oxychloride cement, tetraethyl silicate and silicone paints and their advantages and disadvantages. Paint Manuf. **18**, 84-87 (1948).

SILICONES. This brief discussion treats in an easily understandable form the production of silicones, their use in water-repellent films, silicone rubbers, silicone oils and cross-linked silicones. R. L. Smith. Paint Manuf. **18**, 88-89 (1948).

FUME SCRUBBER FOR LINSEED OIL. Directions on the construction details, scrubbing action, and throughput of linseed oil fumes in a commercial scrubber are given. Paint Manuf. **18**, 90 (1948).

PRACTICAL APPLICATION OF THE GARDNER MOBILOMETER. A digest is given of a paper dealing with a series of experiments made to establish the relationship between paint composition, consistency and yield value as determined by the Gardner Mobilometer, and film thickness obtained on brushing. H. L. Matthijsen. Verfkroniek **20**, 199-202 (1947). (Abstr. through Paint Manuf. **18**, 91 (1948).

PAINTS AND PRINTING INKS. In a paper presented by Mr. W. E. Wornum before the London Section of the Oil & Colour Chemists Association, paints and printing inks were generally discussed as to definitions, effects of tack, letterpress machines, lithographic inks, and film properties. Paint Manuf. **18**, 93-95 (1948).

GUIDE TO PIGMENTS AND RELATED PRODUCTS. The first portion of a glossary of terms used in the paint and varnish industries which is to be published serially, includes terms from Acid to Anhydrous. J. H. Martin & W. M. Morgans. Paint Manuf. **18**, 98-99 (1948).

PAINTING OF ALUMINUM. An exhaustive discussion is given of the physicochemical pro-

perties of paints to be used for protective and decorative purposes on light metals, i.e., adhesion, flexibility at different temperatures, hardness, resistance to heat and humidity, chemical resistance, absence of odors and tastes, noninflammability. Preparation of surfaces, degreasing, electrolytic cleaning, anodic oxidation, primers and methods of application of paints are described in detail. For proper selection of primers, the expansion coefficient of the two materials should be as close as possible. Coefficients for some resins and metals are given. J. J. Meynis de Paulin. Rev. aluminum **24**, 309-17 (1947). (Abstr. through Chem. Abs. **42**, 1744 (1948).

CONDENSATION OF PHENOL WITH DRYING OIL. Phenol is condensed with linseed oil and methyl esters of linseed oil in the presence of oxalic acid, hydrogen chloride, sulfuric acid, and phosphorus pentoxide as catalysts. Maximum condensation with 3% oxalic acid based on phenol, 6 hours, refluxing and 2 hrs open heating at 250 degrees. Ethylenic linkages in linseed oil are attacked. Sulfuric acid catalyzes same reaction at 100 degrees. Gaseous hydrogen chloride causes interesterification, and phosphorus pentoxide is not active below 250 degrees. There are similar results with concentrated sulfuric acid and phosphorus pentoxide at 250 degrees and methyl esters of linseed oil; ethylenic linkages are attacked. P. A. Doshi and T. N. Mehta. J. Sci. Ind. Research **6B**, No. 5, 63-6 (1947), (Abstr. through Chem. Abs. **42**, 1434 (1948).

SOME APPLICATIONS OF ULTRAVIOLET SPECTROPHOTOMETRY IN DRYING OIL RESEARCH. An alkaline-solution technique has been developed permitting the study of the drying and aging process of an oil film using the ultraviolet spectrophotometer. This tchnique has been found to be a valuable tool for investigating some of the chemical changes which occur as a vegetable oil film dries and ages. The drying or autoxidation of an oil film has been shown to proceed along the general lines of a three-stage reaction with the short initial stage being characterized by an accumulation of conjugated diene and triene structures which reaches a maximum at the time the film has set up or becomes continuous. The second or curing stage was characterized by a slow decrease in the measurable conjugated diene and triene structures. The third or deterioration stage was not investigated; however, this method should be readily adaptable to studies involving film breakdown as represented by such phenomena as after yellowing since the development of absorption peaks characteristic of linolenic ketones, for example, would permit an early prediction of yellowing long before the concentration of chromophore groups became great enough for visual observation. Sixteen references. M. H. Hendrickson, R. P. Cox and J. C. Konen. J. Am. Oil Chemists Soc. **25**, 73-77 (1948).

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FOREIGN PATENT DEVELOPMENTS

WATER-REPELLENT COATING MATERIALS. Corning Glass Works. Brit. Pat. 588,762. Applied for May 26, 1944; accepted June 3, 1947. Materials such as glass, glazed or enameled articles, porcelain, asbestos, quartz, mica, etc. are coated with a solution of a non-resinous organosilicon compound, followed by heating to evaporate solvent and convert the organo-silicon compound to a polymerized siloxane. Examples of suitable compounds include methyltriethoxysilicane, dimethyldiethoxysilicane, octadecyltrichlorosilicane. The process may also be applied to other materials such as cotton, silk, rayon, wool, hemp, flax, paper, wood, regenerated cellulose, cellulose derivatives, vinyl polymers, nylon and natural and synthetic plastic materials in various forms.

DRYING OIL-SOLUBLE VINYL RESINS. F. Groff (assigned to Carbide and Carbon Chemicals Corp.). Brit. Pat. 588,836. Applied for Sept. 9, 1944 (applied for in U. S. Nov. 4, 1943); accepted June 4, 1947. Polyvinyl acetal resins are solubilized to enable their incorporation in drying oil materials (e.g. linseed or tung oil) by heating the resins with a water-insoluble carboxylic acid (such as rosin, or oleic, stearic, ricinoleic or abietic acids) to effect an esterification of the free hydroxyl groups of the vinyl resin, and then heating this product with a drying oil. The esterified resins are also compatible with modifiers such as rosin, lac, ester gum, soya bean oil or oiticica oil. Resins used include polyvinyl partial acetals with propionaldehyde, butyraldehyde or valeraldehyde.

SYNTHETIC DRYING OIL. D. E. Adelson and H. F. Gray, Jr. (assigned to Shell Development Co.). Brit. Pat. 589,650. Applied for Aug. 16, 1944 (applied for in U. S. Aug. 11, 1943); accepted June 26, 1947. Polyallyl type alcohols are heated with an unsaturated acid to give synthetic polyallyl esters which are mixed with conventional siccartives, pigments etc. The polyallyl alcohols may be obtained by hydrolysis or alcoholysis of polyallyl esters (e.g. allyl acetate polymers). Or the polyallyl ester may be reacted directly with the unsaturated acid by transesterification. Examples of the unsaturated acid are palmitoleic, oleic, linoleic, linolenic, arachidonic, clupanodonic, eleostearic and licanic acids.

SYNTHETIC RESIN COATING COMPOSITIONS. G. W. Stockton (assigned to Joseph Sankey and Sons, Ltd.). Brit. Pat. 589,654. Applied for Sept. 19, 1944; accepted June 26, 1947. A resinous base for a coating is made by preparing a resole or "A stage" phenol-formaldehyde resin (such as that obtained by reacting an alkyl or aryl substituted phenol with formaldehyde to the state at which a fusible intermediate condensate soluble in the usual solvents results), heating this condensate with about twice its weight of a fatty oil (e.g. a medium linseed stand oil), and adding a small amount of cobalt acetate and lead acetate. This mixture is emulsified and used to coat steel laminations for electrical appliances, and is claimed to have high anti-rust properties, higher electrical resistance than natural varnish and thereby an improved space factor in electrical machines and appliances.

RESINOUS CONDENSATION PRODUCTS. H. S. Lilley (assigned to Imperial Chemical Industries, Ltd.) Brit. Pat. 590,000. Applied for April 9, 1945; accepted July 4, 1947. Methylenequinone compounds, e.g. an oil-soluble alkali-condensed phenolic resin polymer) is heated with an aromatic vinyl compound (e.g. styrene) in the presence of a catalyst (stannic chloride or borofluoroacetic acid) to produce a composition useful in the preparation of quick-drying varnishes or pigmented coatings. An example of a methylenequinone is the product obtained by reacting formaldehyde with m-4-xylenol to give 2-hydroxymesityl alcohol which is then converted to the corresponding orthoquinone methide by conversion to the chloride followed by treatment with alkali.

HOT-MELT COATING MATERIAL. W. E. F. Gates (assigned to Imperial Chemical Industries, Ltd.) Brit. Pat. 590,052. Applied for April 11, 1945; accepted July 7, 1947. Chlorinated naphthalene is heated with polythene (polymers of ethylene) and the mixture used in the molten state to coat or seal, by one or more dipping operations, articles such as dry cell batteries, cardboard boxes etc.

HEAT-TREATMENT OF VEGETABLE AND OTHER OILS. J. A. Reavell. Brit. Pat. 590,218. Applied for May 12, 1944; accepted July 11, 1947. Oils such as linseed tung, perilla, oiticica, soya bean, fish and dehydrated castor oils are polymerized by subjecting them to the action of infra-red generators emitting rays in the wave-length range 10,000 to 15,000 Angstroms.

PROTECTING METALS AGAINST CORROSION. Westinghouse Electric Corp. French Pat. 919,414. Applied for Dec. 26, 1945; delivre Nov. 25, 1946. Metals are coated with an aqueous solution containing disodium phosphate and a compound of titanium such as the tetrachloride, trichloride, hydroxide, nitride, double salt with potassium oxalate, and of less suitability the carbide, dioxide, double potassium fluoride etc. The coated metal is then heated to dryness at about 80° C.

COATING METALS WITH POLYTHENE. Imperial Chemical Industries, Ltd. French Pat. 919,810. Applied for Oct. 31, 1945 (applied for in Great Britain Sept. 1, 1944); delivre Dec. 16, 1946. Metals are coated with polythene by first applying to the metal a substance such as sodium silicate or phenol-formaldehyde, then with a paste of thickened starch of sufficient film thickness so that the first material does not come through, and finally with molten polythene.

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